Quality Assurance Plan

Mammoth Cave National Park
Water Quality Laboratory
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Quality Assurance Plan

For
Mammoth Cave National Park
Water Quality Laboratory
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_____	_____
Laboratory Manager	Date
_____	_____
Quality Assurance Officer	Date

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Laboratory Quality Policy Statement

Mammoth Cave Water Quality Laboratory is dedicated to providing water analysis services both expediently and accurately. Accurate results are necessary for valid research and the continued monitoring of water quality. To assure the accuracy and defensibility of results, there must be written procedures followed in all cases. The following quality assurance plan describes sample handling and quality control procedures, analysis and data reporting methods, as well as, instrument maintenance, and security measures used within the laboratory. All samples entering the laboratory will be analyzed according to the guidelines described in this report.

The **general principles** of our quality assurance program are:

1. Properly tracking the movement of the sample through the laboratory. We will have documentation available to account for the sample location at all times from the moment it enters the laboratory to the time of its disposal.
2. Analyzing samples using standard methods. The samples entering the laboratory will be analyzed according to the methods discussed in this quality assurance plan or a comparable method.
3. Analyzing quality control and performance evaluation standards to check method performance. Quality control procedures and guidelines for acceptance are supplied in this quality assurance plan and will be followed.
4. Using only quality standards and reagents obtained from commercial vendors to complete the analysis of samples.

5. Performing proper instrument calibration and maintenance so that the instrument will be able to generate accurate data with little downtime.
6. Keeping good records that document everything done in the laboratory.
7. Guarding samples, records and instruments from tampering by keeping the laboratory securely locked and inaccessible to non-authorized personnel.

Laboratory Organizational Structure

Personnel Files

All laboratory personnel have files located in the laboratory that contain the name, resume, title and/or job description, educational background and specialized training of the individual. These files are kept to guarantee that the individual doing a job is qualified.

Personnel Training

Only trained personnel will be permitted to analyze samples. Personnel in the process of being trained will analyze samples under the supervision of trained personnel only. Personnel are considered qualified to analyze a sample once they have been educated about the operation of the instrument, method(s) used to analyze the sample, proper quality control measures, have successfully analyzed a sample under supervision with reproducible results and have analyzed a sample of known concentration accurately.

Definitions

Laboratory Supervisor - this individual is responsible for directing the analysts' work, ensuring that there is adequate equipment and supplies to perform analysis, submitting results to clients, making sure that all personnel analyzing samples are qualified and correcting problems when they occur.

Quality Assurance Officer - this individual is responsible for the quality of the results produced in the laboratory by interacting with analysts and the supervisor to make sure that quality control procedures are implemented into good laboratory practices. The quality assurance officer updates the quality assurance plan, monitor's laboratory data, conducts laboratory audits and communicates with accreditation agencies.

Sample Custodian - this individual is responsible of incoming samples and should be able to provide documentation tracking the sample's movement through the laboratory. This individual is also in charge of sample disposal following the analysis.

Analyst - this individual is in charge of analyzing samples that come into the laboratory under the direction of the laboratory supervisor and utilizing proper quality control procedures.

The organizational structure of the laboratory is shown in Figure 1.

Laboratory Facilities

General

It is important that laboratory facilities are maintained in high quality that accurate testing can be safely performed. Laboratory facilities will be clean, safe, secure, and contain all equipment, instruments, reagents, etc. necessary to perform analysis.

Cleanliness

All laboratory areas will be kept clean and free of clutter. Tabletops dirty from spilled samples can make the accuracy of results questionable and areas cluttered with excess papers and materials could contribute to loosing important documents.

Safety

The laboratory is equipped with a fume hood, sink, first aid kit, safety shower, a fire extinguisher and sprinkler system. Broken glass and waste materials have a designated and clearly labeled area in the laboratory. MSDS sheets are kept in house for all chemicals used in the laboratory.

Security

The laboratory facility must be secure at all times; otherwise, it is possible that the accuracy of results could be compromised by sample contamination or that important documents and records could by altered or destroyed. The laboratory area is locked and/or protected by a security system at all times and is accessible only to authorized personnel. In addition, samples are kept in a locked refrigerator prior to analysis.

Sample Handling Processes

Sample Collection

Our laboratory will be both collecting samples for analysis and receiving samples that have been collected by outside clients. Our laboratory is not responsible for the quality of samples collected by other agencies. If a sample entering the laboratory is found to have some problem which may effect the quality of the results (ex. sample bottles that would not protect photosensitive analytes from light or a temperature that would not sustain the integrity of the sample) the problem will be noted on the chain of custody agreement, in the analyst logbook and/or in the analytical report submitted to the client.

When laboratory and/or MACA personnel collects anion and cation water samples, they will be collected in plastic bottles, stored on ice until returned to the laboratory, kept at a temperature of less than 4^{o}C and held for no longer than 28 days as specified by EPA method requirements. The cations will be acidified using sulfuric acid to a pH of ~ 2. Transition metals are collected in plastic bottles, preserved with nitric acid, held at a temperature of less than 4^{o}C and are analyzed within 6 months. Pesticide samples are collected in glass amber vial, buffered using ammonium acetate and analyzed within 14 days. Total Suspended Solids (TSS) samples are collected in plastic bottles, held at a temperature of less than 4 ^{o}C and analyzed within 7 days. When the laboratory is collecting total organic carbon (TOC) samples, the glass bottles are first prepared by rinsing them with dilute nitric acid, covering with aluminum foil and baking for 1 hour at 400^{o}C; the samples will be kept at. Samples that can not be examined immediately will be preserved by holing at a temperature of less than 4 ^{o}C with minimal exposure to light and atmosphere. Acidification with phosphoric or sulfuric acid to a pH equal or less than 2 at the time of collection is especially desirable for unstable

samples and may be used on all samples; however, acid preservation invalidates any inorganic carbon determination on the samples. Therefore, in most cases non-purgable organic carbon (NPOC) will be determined. Chlorophyll A samples are collected in two liter amber plastic bottles that have been pre-rinsed with acetone. The samples are immediately filtered through glass fiber filters (GFF) upon entering the laboratory. The GFF are sealed in clean petri dishes, wrapped in aluminum foil and stored in the freezer up to 3 weeks prior to analysis, according to the standard method requirements. Fecal coliform samples are collected in sterile plastic bottles and/or whirl-pak bags, filtered though membrane 0.45 μm filters and plated into sterile absorbent pads that are prepared with 2.0 mL M-FC broth within 8 hours of collection pre standard method 9222D. Turbidity samples are collected in plastic bottles, kept at a temperature of less than 4 ^{o}C, and analyzed as soon as possible. Fluorescence samples are collected in glass amber bottles that have been rinsed with dilute HCl and pure water. They are refrigerated until analysis and analyzed within 72 hours.

Sample Tracking

A laboratory must be able to produce scientifically valid data that is reliable and can sustain legal questioning. To do this, it is vital that the integrity of the sample be preserved. Sample integrity is accomplished by:

1. Securing the laboratory location so that the sample is in laboratory personnel's physical possession, in view of laboratory personnel, secured to prevent tampering, or in a secure location inaccessible, except to authorized personnel.
2. Keeping chain of custody records that track the movement of the sample between field and laboratory settings. Each chain of custody record contains the sample ID, laboratory ID, name of sampler, analysis requested, client name, date and time of

sample collection, sample custodian signature, signature of the individual(s) relinquishing the sample, composition of the sample. The chain of custody agreement used by the laboratory is shown in Figure 2.

3. Assigning a laboratory identification (ID) number every sample upon entrance into the laboratory.
4. Samples are stored in the refrigerator at a temperature of less than 4 ^{o}C to ensure the integrity of the sample. Standards are kept in a separate refrigerator from samples. This notebook has the laboratory ID, sample ID, client name, date the sample was collected, date the sample was received, date the sample was analyzed, analysis requested, and the initials of the individual who received and analyzed the sample. The laboratory login / logout book is shown in Figure 5.
5. Keeping track of sample disposal. Following the analysis of the sample and review of resulting data, the sample is disposed of down the drain. Only regular samples composed of natural river, spring, creek, etc water will be poured down the drain. No samples containing harmful components will be dumped down the drain. Samples containing harmful components will be discarded into a waste container. The amount and nature of the waste inside the waste bottle is noted on the waste bottle. The laboratory I.D. number is highlighted in the laboratory login/logout book to indicate that the sample was discarded.

Problems With Samples

If, at any time, it is discovered that there might have been a problem with the integrity of a sample after entering the laboratory, the customer, the quality assurance officer and the laboratory supervisor are notified immediately and the problem is addressed. Possible

solutions to problems with sample integrity could be reanalyzing another freshly collected sample or correcting problems with sampling conditions or holding methods.

Sample Holding Times

Anion and Cation samples are analyzed within 28 days according to the method. Transition metals samples are analyzed within 6 months. Pesticide samples are analyzed within 14 days. TSS samples are analyzed within 7 days. TOC samples are analyzed as soon as possible and are held for no longer than 28 days. Fecal coliform samples are within 8 hours of collection and analyzed 24 ± 2 hours from time of filtering. Chlorophyll A samples are immediately filtered through glass fiber filters and analyzed within 3 weeks. Turbidity samples are analyzed as soon as possible. Fluorescence samples are analyzed within 72 hours. Samples coming into the laboratory, whose holding times have expired, will still be analyzed. However, the fact that the holding times for the samples had expired at the time of the analysis will be included in the sample login/logout book and in the report issued to the clients. All samples are kept for at least 30 days after the analysis date.

Waste Handling

Dangerous wastes are put into clearly labeled waste bottles. How much waste, the type of waste and who disposed of the waste are recorded on a sheet that accompanies the waste bottle. Once filled, the waste bottle is collected by Pollution Control Industries, Inc (phone: 1-800 228-8845) during regularly scheduled visits to the park.

Analytical Procedures

General

Standard methods for analysis are created to ensure that all samples are analyzed correctly and that results generated are accurate. Standard methods have been tested repeatedly and have been proven reliable over time. Accepted and/or applicable methods are used to analyze all samples. The analytical procedures described in this section are to be used to analyze samples.

Glassware Cleaning Procedure

Much of the equipment used in the laboratory is for single use and disposable. However, if glassware is to be reused it must be cleaned properly to prevent contamination of subsequent samples. Laboratory glassware will be cleaned in the following manner.

1. Wash glassware with hot tap water and detergent.
2. Rinse with hot tap water.
3. Rinse three times with deionized water utilizing a NANOpure® Infinity filtration system.
4. Allow bottles to air-dry on drying rack, except for methods requiring acid or acetone rinse and/or oven drying, as required by the method.

**No class A volumetric glassware will be baked.

Reagent Water Preparation

Water must be free from contamination that could interfere with results. For this reason, the laboratory water is purified using a Barnstead NANOpure® Infinity filtration system, model number D8971. The purity of the water is checked before analysis by analyzing reagent water blanks.

Standards

All chemicals and reagents used in the course of the analysis of samples are ordered from reputable commercial vendors and are guaranteed pure. Certificates indicating this purity accompanying the standards are kept in a three ring binder.

Methods

1) *Fluoride*, *Chloride*, *Nitrite*, *Bromide*, *Nitrate*, *Phosphate* and *Sulfate* are to be analyzed using an ion chromatograph from Dionex by EPA method 300.1: Determination of Inorganic Anions in Drinking Water By Ion Chromatography.
2) *Lithium, Sodium, Ammonium, Potassium, Magnesium and Calcium* are to be analyzed using an ion chromatograph from Dionex using a method developed by that company.

   This method uses a Cation Atlas Electrolytic Suppressor, an IonPac CG12A-5 um guard, an IonPac CG12A-5 um column with a flow rate of 0.5 ml/min, a sample loop of 25 uL and dilute methanesulfonic acid solution as the eluent. According to this method samples are collected in plastic bottles and preserved until the time of analysis using sulfuric acid. The samples are kept in a refrigerator away from open standards and held for no longer than 28 days before analysis.
3) *Iron II, Iron III, Copper, Cadmium, Manganese, Cobalt, Zinc and Nickel* are to be analyzed using an ion chromatograph from Dionex using a method developed by that company.

   This method uses an IonPac CG5A 4 x 50 mm guard and an IonPac CS5A 4 x 250 mm column. MetPac PAR Postcolumn Reagent Diluent supplied by Dionex is used to make the postcolumn reagent and MetPac PDCA Eluent Concentrate is used to make the necessary eluent. The eluent flow rate for the method is 1.2 mL/min, the post

column reagent flow rate is 0.6 ml/min. The sample loop is 50 μL. According to this method samples are collected in plastic bottles and preserved until the time of analysis using nitric acid. The samples are kept in a refrigerator away from standards and held for no longer than 6 months before analysis.

4) *Cyanazine, Simazine and Atrazine* are to be analyzed using an ion chromatograph from Dionex using a method developed by that company.

   This method uses an OmniPac PCX-500 4 x 250 mm analytical column with an OmniPac PCX-500 4 x 50 mm guard. The method requires an acetonitrile-water mixture with a sodium phosphate buffer as the eluent, a flow rate of 0.7 mL/min and a 100 μL sample loop.

5) Total Organic Carbon analysis will be performed using a TOC-V CSH/CSN from Shimadzu according to method 5310 B High Temperature Combustion Method, Standard Methods for the Examination of Water and Wastewater.

6) Total Suspended Solids analysis will be performed according to the Standard Methods for the Examination of Water and Wastewater method 2540 D.

7) Fecal Coliform analysis will be performed according to method 9222 D. Fecal Coliform Membrane Filter Procedure Standard Methods for the Examination of Water and Wastewater.

8) Chlorophyll A analysis will be performed according to EPA method 445.0 In Vitro Determination of Chlorophyll A and Pheophytin A in Marine and Freshwater Algae by Fluorescence.

9) Turbidity analysis will be performed according to the Standard Methods for the Examination of Water and Wastewater method 2130.

10) Fluorescence analysis will be performed using a method titled Spectrophotometric Discrimination of River Dissolved Organic Matter.

This method requires samples to be collected in glass amber bottles rinsed with dilute HCl and water prior to sampling and to be stored in the refrigerator for a period of no longer than 72 hours before analysis. A spectrophotometer capable of analyzing samples with low concentrations (approx 0.1 mg/L) must be used to analyze the samples.

Copies of these methods will be stored in house and available to all personnel.

Quality Control Procedures

General

Even standard methods used by a trained analyst using instruments that are in good working condition are not completely error proof. Therefore, laboratory work must be checked periodically and reaffirm the accuracy of reported results. This is achieved by following quality control procedures that check the validity of the method(s), condition of the instrument(s), and the purity of the reagent water.

Definitions

Accuracy - A measure of how close a measured value is to the true value. Assessed by means of percent recovery of spikes and standards.

Calibration Standard (CAL) - A solution prepared from a stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

Continuing Calibration Verification (CCV) - A mid-range calibration standard that is analyzed periodically during sample analyses.

Laboratory Duplicate - Two solutions obtained from the same sample bottle and analyzed separately using the same technique. This procedure helps to determine the amount of precision associated with the results.

Laboratory Fortified Blank (LFB) - An aliquot of reagent water, acidified if necessary, that matches the matrices of the sample and standards, to which a known quantity of each method analyte is added in the laboratory. The LFB is analyzed exactly like a sample and its purpose is to determine whether the method is within accepted control limits. Sometimes these samples are referred to as "spiked blanks".

Laboratory Reagent Blank (LRB) - An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents or apparatus.

Method Detection Limit (MDL) - The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

Performance Evaluation Sample - performance evaluation sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified by the supplier. The true value of the concentration of the reference material is unknown to the laboratory at the time of the analysis.

Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is usually expressed in terms of standard deviation.

Quality Control Sample (QCS) - A solution containing a known concentration of each method analyte derived from externally prepared test materials. The QCS is obtained from a source external to the laboratory and is used to check laboratory performance. This is sometimes called an Initial Calibration Verification (ICV).

Surrogate - An analyte that is extremely unlikely to be present in the sample and is added to the sample in a known concentration and measured using the same procedures used to measure the other analytes in the sample. This quality control measure checks instrument performance and analyst technique.

Standard Procedure

Most methods have accompanying quality control procedures to be followed. In the event that these measures differ from the following standard laboratory quality control measures, the more stringent quality control protocol is used. In general, however, the standard laboratory quality control measures are as follows.

To check the **quality of the reagent water** being used, a laboratory reagent blank is analyzed every 10-15 samples. The water will be considered acceptable if the resulting concentrations for the analytes are less than the method detection limits

To check the **calibration curve** being used, one calibration check is run per every 15-20 samples analyzed or at the end of each batch of samples. This check can be either a LFB of a concentration close to that of the analytes in the sample or a quality control standard. If a LFB is used, the curve will be considered acceptable if 80-120% recovery is obtained. If a quality control sample is used, the curve will be considered acceptable if the results meet the acceptability requirements supplied for the sample by the vendor.

To check the **accuracy of the method**, one LFB of known concentration is analyzed per 15-20 samples analyzed. An acceptable percent recovery will be between 75 and 125% for each LFB. Also, a laboratory fortified sample matrix sample will be analyzed per 15-20 samples analyzed. The results of this analysis will be considered acceptable if the percent recovery is 75-125%.

To check the **precision of the method**, one laboratory duplicate will be analyzed per every 10 samples. The method will be considered precise if the percent difference is ± 10%

General Quality Control procedures are also presented in Table 1 for easy reference.

In accordance with water quality procedures the laboratory will analyze one performance evaluation sample annually and one quality control sample quarterly. The performance evaluation sample is deemed acceptable if the vendor providing the sample affirms that the results were acceptable. The quality control sample results are deemed acceptable if the results fall within the acceptability requirements supplied by the vendor.

Equations

$$\text{Percent Recovery} = \frac{\text{Amount of Analyte or Surrogate Found}}{\text{Amount of Analyte or Surrogate Added}} \times 100\%$$

$$\text{Percent Difference} = \frac{(\text{Conc 1} - \text{Conc 2})}{[(\text{Conc 1} + \text{Conc 2})/2]} \times 100\%$$

Method Detection Limits

Method Detection Limits (MDL) will be calculated according to the procedure outlined in 40 CFR 136 Appendix B or according to the procedure specific for a particular method, if one is given. The laboratory will achieve MDL(s) at or below those given for each specific parameter. Current MDLs for the laboratory are given in Table 3. MDLs are recalculated annually or as specified by the method(s).

Instrumentation

General

In order to analyze samples the analysts must rely on their instruments. This dependency means that there must be methods to monitor the instrument, and there must be measures to prevent problems with instrument performance, as well as fix any problems that occur.

Instrument Calibration Procedures

Before the instruments can be used to accurately analyze a sample it must be calibrated. Calibration of the instrument to be used requires by making a minimum of a 3 point calibration curve consisting of high and low concentration values and at least one other concentration evenly divided between these values. The computer software calculates the relative standard deviation (RSD) for the curve. If the RSD is less than 15% and the continuing calibration check samples are within acceptable limits the calibration curve is considered acceptable.

If a sample is analyzed and found to have a concentration of some analyte that falls outside the calibration curve range, either the sample is diluted and reanalyzed or a new calibration curve (one that encompasses the analyte concentration of the sample) is made. A new calibration curve is made with every sample run for turbidity, anions, cations, transition metals, triazines, and total organic carbon analysis. Chlorophyll-a calibration curves are recalculated every 6 months. Fecal coliform analysis is accomplished through a counting process and TSS analysis is performed using weight differences; therefore, neither require a calibration curve. Fluorescence analysis is only a qualitative process and does not use a calibration curve.

Preventive Maintenance

Preventive maintenance is a term used to describe actions taken to assure that the instruments remain in good condition. This is beneficial because of the resulting decrease in instrument downtime and the lessened cost of instrument repair. Measures to assure good instrument performance are supplied in the manuals given by the manufacturers. These manuals are kept with other documents and notebooks in the laboratory and are easily available to personnel for reference. Some preventive maintenance procedures for the laboratory are given in Table 2.

Service Agreements

Despite proper maintenance, it is still possible for instruments to need repair. For this reason, the laboratory has service agreements with the manufacturers of the instruments to handle any necessary repairs.

Instrument Downtime

If an instrument used to analyze a sample is not working properly and will not be able to be repaired in time for a sample to be analyzed within its holding time, the client will be notified.

Maintenance Log

An Equipment and Instrument Maintenance Log book is kept in a three ring binder in the laboratory. In this logbook, a record is kept of when the instrument was not operational, what problems the instrument had, what was done to correct these problems and when the instrument was considered operation again. Also, service agreements with the manufacturers of our instruments and receipts for any parts delivered are kept in this book.

Data

Raw Data

Upon analysis of the sample, the instrument generates the initial data on the sample. To better improve efficiency and accuracy, calculations are done by the instrument. When utilizing the IC, TOC analyzer and the Nephelometer, the instruments use calibration curves and the peak areas to output an actual concentration. For analysis not performed by an instrument, such as Fecal coliform and TSS analysis, raw data is in the form of numbers of colonies per 100 mL of water and differences of measured weights, respectively.

Data Validation

Before any data is submitted to a customer, the analyst reviews all quality control procedures corresponding to the analysis of that particular sample. The analyst verifies that the sample integrity had been preserved up until the time of analysis and that proper quality control tests and instrument checks were performed within acceptable results. If the analyst has a question about any of the previous they contact either the laboratory supervisor or the quality assurance officer. They will then review all the materials and certify that the data is accurate and can be released to the customer.

Data Reporting

Once the data is deemed acceptable by the analyst, laboratory supervisor, and/or quality assurance officer, a standard analytical report is issued to the client. This report is shown in Figure 4. A copy of this report is kept in the project files.

Corrective Action

General

Corrective action must be taken upon the unacceptable analysis of a performance evaluation sample or a quality control sample.

Standard Procedure

Once the unacceptable sample is analyzed, the problem is noted in the equipment and instrument maintenance log. At that time, no other samples will be analyzed until the problem is corrected and a new entry in the manual indicates that a new quality control or performance evaluation sample has been analyzed acceptably. At the discretion of the quality assurance officer, a sample may be analyzed for a particular analyte if the test for that analyte was acceptable. For example, if the quality control sample was unacceptable for chloride, but was acceptable for fluoride, the quality assurance officer can permit the analysis of a sample for fluoride.

Many times the source for an unacceptable result could be human error; for this reason, a new quality control sample is analyzed first. If the second quality control sample remains unacceptable, the laboratory supervisor and quality assurance officer are notified of the problem. They attempt to discover the source of the problem, checking the water, standards, preparation methods and instruments. Once they have found the problem, corrected it and successfully analyzed a quality control sample, the analyst notes the cause of the problem, corrective action taken and affirms that the laboratory is again able to accurately analyze the sample in the equipment and instrument maintenance log.

If a performance evaluation sample is analyzed with unacceptable results a follow up analysis is performed immediately.

Record Keeping Practices

General

Good record keeping practices are vital to a laboratory's quality program. Keeping good records allows anyone at anytime to review what the laboratory has done in the course of analyzing a sample. Our laboratory will keep detailed and organized records of all procedures done in the laboratory. All written records will be done in ink. If changes need to be made to records at anytime, a single line will be drawn through the incorrect information. The correct information along with the initials of the individual making the change will be recorded above the previous information. These records will be available to all clients and certifying authorities that wish to review them.

Laboratory Notebooks

Quality control data such as LFBs, LRBs, quality control standards, and performance evaluation samples will all be kept on file in the laboratory. The date and time will be written on all these documents for reference and organization purposes. Data kept in bound notebooks such as the analyst's notebooks containing information about each analysis, refrigerator login/logout books and cumulative sample information book will be kept in the laboratory at all times.

Project files will be kept in the laboratory. These project files will contain the chain of custody agreement along with any other paperwork regarding the shipping of the sample, analysis results report from the instrument, and a copy of the analysis report that was given to the clients. All data on every sample is kept for a minimum of ten years for reviewing purposes.

Computer Files

In addition to keeping a hard copy of data, the laboratory will also keep computer files of analysis results either on the computer or on computer disks or CDs and/or network servers for backup purposes. Files kept on a computer will be accessible to authorized personnel only and secured by a password. Disks and CDs are kept with hard copies of documents and are secure inside the laboratory.

Figure 1 Laboratory Organizational Chart

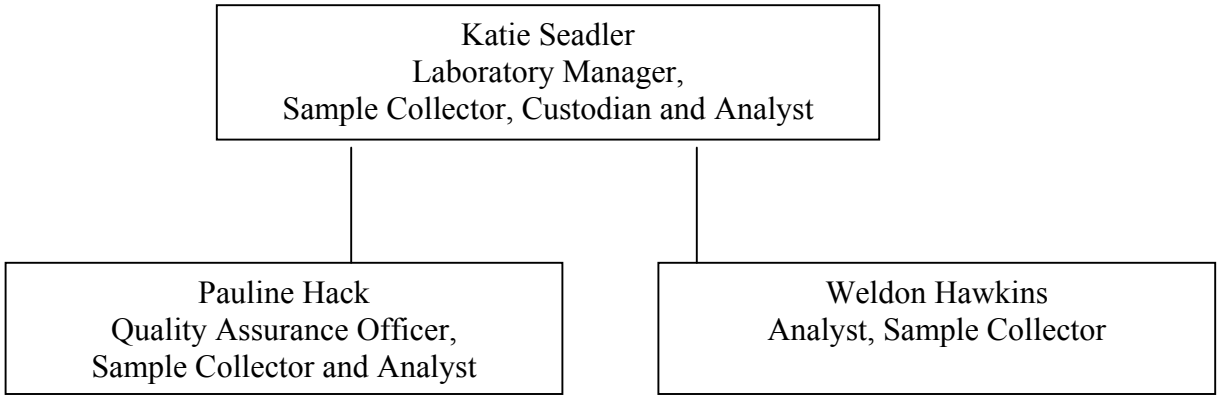

Figure 2 Chain of Custody Record

Sampling Method:

Chain of Custody Record

Mammoth Cave National Park
Water Quality Laboratory
NPS Warehouse, Maintenance Road
Mammoth Cave, KY 42259
270-758-2152, fax: 270-758-2663

Sampler's Name:	Collection Date/Time	
Released by:	Date/Time	Received by:
Released by:	Date/Time	Received by:

Client Name, address, phone, contact person:			Sample Date/Time	Solid, liquid, or sludge, dye receptors	Preservatives Used	Sampling Method grab (G) composite (C)	Project / Site Description:
Laboratory I.D. *(Laboratory use only)*	Sample I.D.	Analysis Requested					Additional Information:

Figure 3 Laboratory Collection Label

Sample ID:	Date:
Sampled By:	Time:
Location:	
Analysis:	
Comments:	

Figure 4 Laboratory Report

Mammoth Cave National Park
Water Quality Laboratory
Mammoth Cave, KY 42259
270-758-2152 Fax: 270-758-2663

Customer Sample ID: Sample Date:

Customer Name: Injection Volume:

No.	Analyte	Date Collected	Date Analyzed	Sequence Name	Dilution Factor	Amount (ppm)	Detection Limit (ppm)

nd = None Detected
NA = Not Analyzed
BDL = Below Detection Limit
BQL = Below Quantification Limit

Comments:

Submitted by: _____
Kathryn J. Seadler, Hydrologist
Katie_Seadler@nps.gov

Figure 5 Laboratory Login/Logout Book

Lab ID	Sample ID	Sample Name	Sample Date	Client Name	Date/Time Received	Received By	Analysis Requested	File/ Sequence Name	Date of Analysis	Analyzed By	Reported to client	Additional Notes or Information

Table 1 General Quality Control Procedures

Quality Control Procedure	How Often	Acceptability Range
Laboratory Reagent Blank	1 in every 20 Samples	Lower than the method detection limit
Continuing Calibration Check	1 in every 10 Samples	80-120% recovery or acceptability range specified by vendor
Laboratory Fortified Blank	1 in every 20 Samples	75-125% recovery
Laboratory Fortified Sample Matrix	1 in every 10 Samples	75-125% recovery
Laboratory Duplicates	1 in every 10 Samples	± 10% Difference
Quality Control Sample	Quarterly	Acceptability range specified by vendor
Performance Evaluation Sample	Annually	Acceptability range specified by vendor

Table 2 Preventive Maintenance

TOC Instrument Checks	How Often	Corrective Procedure
Check Dilution Water	Daily	Add Sufficient Amount of Water
Check Acid	Daily	Add Sufficient Amount of Acid
Check Drain Vessel Water Level	Daily	Add Sufficient Amount of Water
Check Humidifier Water Level	Daily	Add Sufficient Amount of Water
Check Printer Chart Paper	When Indicator Light Comes On	Add More Paper
Catalyst Regeneration	Poor Performance	Perform Catalyst Regeneration
Washing Catalyst	Poor Reproducibility or Sensitivity	Perform Catalyst Washing Procedure
Replacing Catalyst	Poor Performance	Replace With New Catalyst
Replacing Carrier Gas Purification Tube	As Needed	Replace the Tube
Check Surface of Combustion Tube	Whenever Catalyst Is Replaced	Wash The Tube To Remove Accumulated Salts
Check Air Cylinder	Two To Three Months	Replace Cylinder
Check Co_2 Absorber	Annually	Replace Absorber
Check Halogen Scrubber	When Absorbent Turns Black	Replace Scrubber
Check Syringe Plunger Tip	If Leaks Are Present	Replace Syringe Plunger Tip
Check Ferrules	If Gas Leaks Are Present	Replace With New Ferrules
Check O Rings	If Gas Leaks Are Present	Replace With New O Rings

Spectrofluorophotometer Instrument Checks	How Often	Corrective Procedure
Check bulb	When good results cannot be obtained	Call Shimadzu; schedule a service call

Ion Chromatograph Instrument Checks	How Often	Corrective Procedure
Eluents	Every month	Remake eluent solutions
Priming pump	With every use	Prime the pump
Check piston seal leaks	Daily	Rinse piston
Chamber and tubing check	Daily	Use dionized water to flush out chemicals
Primary and backup piston seals	Every six months or if a leak develops	Replace seals
Check valves	Whenever pump will not prime	Clean or replace valves
Gasket and o-ring check	As needed	Replace o-ring and gasket

Turbidimeter Instrument Checks	How Often	Corrective Procedure
Lamp check	Whenever not working properly or good results cannot be obtained	Replace or clean Lamp with alcohol
Dilution Water Check	Whenever it is not less than 0.5 NTU	Replace with higher quality water
Calibration Check	When measurements indicate a negative turbity value or good results cannot be obtained	Perform new calibration

Table 3 Method Detection Limits

Analyte	MDL	Analyte	MDL
Fluoride	0.0200 mg/L	Iron II	0.2511 mg/L
Chloride	0.0996 mg/L	Iron III	0.0502 mg/L
Nitrite	0.1015 mg/L	Copper	0.0502 mg/L
Bromide	0.0996 mg/L	Cadmium	0.2517 mg/L
Nitrate	0.0991 mg/L	Manganese	0.0999 mg/L
Ortho-Phosphate	1.9980 mg/L	Cobalt	0.0501 mg/L
Sulfate	0.1000 mg/L	Zinc	0.0502 mg/L
Lithium	0.0250 mg/L	Nickel	0.0996 mg/L
Sodium	0.1000 mg/L	Cyanazine	To be determined
Ammonium	0.1250 mg/L	Simazine	To be determined
Potassium	0.2500 mg/L	Atrazine	To be determined
Magnesium	0.1250 mg/L	Total Suspended Solids	2.5 mg
Calcium	0.2500 mg/L	Fecal Coliform	1.0 col/100 mL
Total Organic Carbon	1.0000 mg/L	Chlorophyll A	0.0820 mg/L
Turbidity	0.001 NTU	Fluorescence	0.1000 mg/L